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DYNAMIC THERMOPHYSICAL MEASUREMENTS IN SPACE

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ABSTRACT

The objective of this research is to develop an accurate dynamic technique which, in a microgravity environment, would enable performance of thermophysical measurements on high-melting-point electrically conducting substances in their liquid state. In spite of the critical need in high temperature technologies related to spacecraft, nuclear reactors, effects of powerful laser radiation, etc., and in validating theoretical models in related areas, no accurate data on thermophysical properties exist. This is primarily due to the limitation of the reliable steady-state techniques to temperatures below about 2000 K, and the accurate millisecond-resolution pulse heating techniques to the solid state of the specimen. The limitation of the millisecond-resolution techniques to temperatures below the melting point stems from the fact that the specimen collapses due to the gravitational force once it starts to melt. The rationale for the use of the microgravity is that by performing the dynamic experiments in a microgravity environment the specimen will retain its geometry, and thus it will be possible to extend the accurate thermophysical measurements to temperatures above the melting point of high-melting-point substances.

The research includes: (1) establishment of the geometrical stability criteria for specimens when heated rapidly, in a microgravity environment, to temperatures above their melting point, (2) development of techniques and instruments for the accurate measurement of quantities, such as current, voltage, temperature, under conditions required for dynamic experiments in a microgravity environment, (3) demonstration of the applicability of the millisecond-resolution pulse heating technique by performing, in a microgravity-environment, definitive measurements of the heat of fusion of a refractory metal such as niobium and tests for heat capacity and electrical resistivity measurements in the liquid phase, (4) exploratory work on the feasibility of measuring thermal conductivity of high temperature conducting and semiconducting liquids, and (5) design and construction of a system for accurate dynamic measurements of thermophysical properties in the Space Shuttle.

1. INTRODUCTION

1.1. Objective

The objective of the research is to develop an accurate dynamic technique which, in microgravity environment, would enable thermophysical measurements to be performed on high-melting-point electrically-conducting substances in their liquid state at temperatures above 2000 K.

1.2. Rationale

Reliable thermophysical properties data on high-melting-point substances in their liquid phase are almost nonexistent due to the serious and often unsurmountable difficulties involved in performing experiments at very high temperatures. Yet, such data are critically needed in various areas of high-temperature technologies related to spacecraft and rocketry (protective heat shield, etc.), understanding the behavior of substances exposed to powerful laser radiation, studies related to the operation of nuclear reactors, as well as in validating the theoretical models and predictions of the behavior of substances under extreme conditions.

Steady-state techniques for the reliable measurements of properties are generally limited to temperatures below about 2000 K. Dynamic techniques involving rapid pulse heating of the specimen have the potential of extending this limit to higher temperatures. Millisecond-resolution techniques have been developed which can provide accurate data up to about 4000 K. Such techniques, however, are limited to the solid phase of the specimen, since the specimen disintegrates and collapses under the gravitational force once it reaches its melting point. It seems logical, therefore, that by performing the experiments in a microgravity environment, it will be possible to extend the limits of the accurate dynamic techniques to temperatures above the melting point of the specimen.

1.3. Summary of Research

The first phase of the work is to establish the geometrical stability of a specimen when heated rapidly to temperatures above its melting point in a microgravity environment. A system has been designed and constructed which

permits rapid heating of the specimen to temperatures above its melting point and checking of the geometrical stability of the liquid specimen. This system has been flown three times on board of KC-135 aircraft. The results suggested several refinements and modifications in the system, which are presently being made. Some additional work is needed in this direction to understand the complete behavior (geometrical stability) of the liquid specimen heated rapidly by the passage of a high current pulse through it, and as a result, optimize the specimen geometry and the operating conditions of the overall system.

The second phase of the work is to add new measurement capabilities to the system and to demonstrate the applicability of the technique by performing definitive measurements of the heat of fusion of a refractory metal, such as niobium, in a microgravity environment. In addition, measurements of heat capacity and electrical resistivity in the liquid phase will also be performed. Exploratory research will be conducted to determine the feasibility of measuring another important property, thermal conductivity, of high temperature conducting or semiconducting liquids utilizing the dynamic heating technique in a microgravity environment.

As much as possible, some of the initial tests will be conducted on board of KC-135 aircraft. However, the ultimate goal is the performance of the measurements in the Space Shuttle for reasons that it provides a better, a more reliable, and a longer microgravity environment essential for the success of all the experiments. For this purpose, a miniaturized and highly automated millisecond-resolution system will be designed and constructed.

2. BACKGROUND

2.1. Measurements on Solids by a Dynamic Technique

The increasingly critical need for thermophysical properties data above the limit of accurate steady-state experiments has motivated the establishment of the Dynamic Measurements Laboratory at the National Bureau of Standards. As the result of the pioneering research conducted in this laboratory, a novel dynamic technique has been developed for accurate thermophysical measurements at high temperatures. During the past decade or so, this laboratory has provided reliable data on selected thermal, electrical, and optical properties

of a number of electrically-conducting solids at high temperatures (1500 - 3700 K), up to their melting points. The measured properties include: heat capacity, electrical resistivity, thermal expansion, hemispherical total emissivity, normal spectral emissivity, temperature and energy of solid-solid phase transformations, and melting temperature.

The technique involves heating the specimen resistively from room temperature to temperatures of interest in short times (typically about 1 s) by passing an electrical current pulse through it; and simultaneously measuring the pertinent experimental quantities with millisecond resolution. Because of the short heating time, this dynamic technique circumvents, to a very large extent, the problems (such as, chemical reactions, evaporation, specimen containment, large heat transfers, etc.) which seriously affect the reliable operation of conventional steady-state techniques at temperatures above about 2000 K. The dynamic technique is limited, however, to measurements on solids up to their melting temperatures, at which point the specimens become geometrically unstable and collapse due (in large measure) to the influence of gravity.

2.2. Measurements on Liquids by Dynamic Techniques

There are two possible options in extending the measurements to temperatures above the melting point of the specimen. These options are: (1) submillisecond heating of the specimen and performance of the measurements with microsecond resolution, and (2) performance of the experiments in a microgravity environment with millisecond resolution.

In the first case, heating of the specimen is very fast in comparison to its movement under the gravitational force; thus, all the pertinent measurements may be performed on the liquid specimen before the specimen collapses. This technique has been used by a few investigators to obtain data on liquid metals. However, there are two major objections to this technique: (1) because of the very high speeds (microsecond resolution) measurements of the experimental quantities are considerably less accurate than those made at slower speeds (millisecond resolution), and (2) because of the very high heating rates, the validity of the assumption that the specimen is under thermodynamic equilibrium at any given instant becomes doubtful and, as a

result, the significance of determined properties becomes questionable.

The second case, namely performing the measurements in a microgravity environment, is very attractive because it is a natural extension of the highly accurate millisecond-resolution technique.

3. Measurements in a Microgravity Environment

3.1. Accomplishments and Present State

A research project, supported in part by NASA, was initiated in our laboratory to investigate the feasibility of performing thermophysical measurements in a microgravity environment utilizing the millisecond-resolution dynamic heating technique. The first step was the design, construction and testing of an equipment package, and performance of preliminary experiments to determine stability of the liquid specimens at high temperatures. For this initial phase, we selected the KC-135 flight program from among several NASA facilities for microgravity research (drop tubes, drop towers, other research aircraft, etc.) on the basis of compatibility with our measurement system requirements: (1) near-zero gravity time of 2 to 5 s duration, (2) short time interval (few minutes) between successive experiments, (3) "hands-on" access to the experiment package by test personnel, (4) flexibility of performing experiments in a "free-float" or "tied-down" mode, and (5) large experiment package (volume $\approx 40 \text{ ft}^3$, weight $\approx 700 \text{ lbs}$).

3.1.1. Test Equipment package

The test equipment package for experiments on board of KC-135 aircraft was constructed as two separate units: (1) the pulse-heating system (nominally 2' wide x 4.5' long x 3.5' high; weight $\approx 450 \text{ lbs}$) which includes the specimen cartridge cell and all measuring and recording instruments; and (2) the main battery pack (nominally 2' wide x 4' long x 1' high; weight $\approx 250 \text{ lbs}$) which supplies the electrical power for pulse heating the specimen.

The instrumentation in the pulse-heating system includes: a single-wavelength pyrometer, a high-speed framing camera, a two-channel digital storage oscilloscope for recording the specimen temperature and the

current through the specimen, and a four-channel time delay generator for controlling the timing of various events such as, triggering the recording system, turning the framing camera on and off, closing and opening two fast-acting relay-driven switches which are connected in series in the power-pulsing circuit, etc. The second switch is closed 100 ms before and opened 100 ms after the first switch and thus serves as a backup in the (unlikely) event the first one fails to open at the end of the heating period. An "oscillo-streak" attachment on the framing camera enables a streak-image of an analog oscilloscope display of the pyrometer signal and the image of the rapidly heating specimen to be simultaneously recorded on the film; this feature greatly simplifies the analysis of the high-speed photographs. The framing camera is powered by a separate pack of gel cells in order to minimize the need for electrical power from sources external to the test equipment package. However, approximately 0.5 kilowatts of 110 V (60 Hz) a.c. power is required to operate certain instruments such as oscilloscopes and timers.

Each specimen is mounted inside its own cartridge cell containing argon gas at a pressure of 1 atm (gauge). The cells are designed so that they can be quickly inserted into and removed from the power-pulsing circuit before and after each experiment. A total of ten identical cartridge cells were fabricated thereby permitting up to ten specimens to be studied during a single KC-135 flight.

3.1.2. Preliminary Experiments on Board of KC-135

Three series of dynamic experiments under microgravity conditions were performed on board of KC-135 aircraft. The objectives were: (1) to test the integrity and operation of the equipment package, and (2) to investigate the behavior of the specimen during melting and postmelting periods. As the result of the experiments, after each flight, additions and changes were made in the equipment and specimen assembly to improve the operation of the system and specimen stability in the liquid phase.

The specimens (niobium) were fabricated in the form of cylindrical rods, 3 mm in diameter and 18 mm in length (distance between clamps). The center portion of each rod was reduced in diameter or "necked-down" in order to define the length of the zone which undergoes melting. The nominal dimensions

of the melting zone, that is, the "effective" specimen, were as follows: a diameter of about 2.5 mm, and an aspect ratio (length:diameter) in the range of 1:1 to 3:1. The portions of the rods near the clamps were tapered to a diameter of 2 mm in order to reduce axial heat conduction from the "effective" specimen. The duration of the current pulse varied, depending on the experiment, between approximately 1.8 and 4.5 s. The corresponding heating rates were in the range 700 to 1700 K•s⁻¹. During pulse heating, the behavior of the specimen was photographed by a high-speed framing camera operating at 500 fps while other pertinent experimental quantities such as the signals from the optical temperature sensor and from the accelerometers were recorded every 5 ms by digital storage oscilloscopes. Analyses of the (surface) radiance temperature-time data revealed that "effective" specimens with aspect ratios of 1.5:1 or less tended to yield the largest excursions (about 50 K) into the liquid phase before becoming unstable.

3.2. Planned Work

Research is in progress to establish optimum configuration of the specimen geometry and optimum operational conditions of the measurement system in order to maximize the temperature excursion of the specimen in the liquid phase. A new design, triaxial configuration for the specimen and current return paths, for the experiment chamber is being investigated. With this new design, it will be possible to balance electromagnetic forces on the specimen, and account for the surface tension of the specimen.

As a step toward thermophysical measurements, research has been initiated to design and construct novel high-speed pyrometers (multiwavelength and spatial scanning) for temperature measurements and instrumentation for pulse electrical measurements. As a part of the verification scheme, measurement of the heat of fusion of niobium has been performed in our laboratory utilizing a recently developed microsecond-resolution technique. This result will be compared with the data on heat of fusion that will be obtained in the first phase of the thermophysical measurements in the microgravity environment. The next step will be measurements of heat capacity and electrical resistivity of liquid niobium. Exploratory work has begun to

determine the feasibility of measuring another important property, thermal conductivity, of high temperature conducting or semiconducting liquids utilizing the dynamic heating technique in a microgravity environment. These measurements are likely to require experiment durations greater than 20 s. Studies of the requirements for the experiments in the Space Shuttle have begun. Upon completion of these studies and the preliminary experiments on board of KC-135 aircraft, design and construction of a highly-automated apparatus for thermophysical measurements in the Space Shuttle will be started.

4. PUBLICATIONS

1. A. Cezairliyan, Dynamic Technique for Measurements of Thermophysical Properties at High Temperatures, International Journal of Thermophysics, 5, 177 (1984).
(This publication is a summary of research performed in the Dynamic Measurements Laboratory at NBS and contains 50 references to publications describing the results of various aspects of our work until 1984).
2. A. Cezairliyan and A.P. Miiller, Heat Capacity and Electrical Resistivity of POCO AXM-5Q1 Graphite in the Range 1500-3000 K by a Pulse Heating Technique, International Journal of Thermophysics, 6, 285 (1985).
3. A.P. Miiller and A. Cezairliyan, Thermal Expansion of Molybdenum in the Range 1300-2800 K by a Transient Interferometric Technique, International Journal of Thermophysics, 6, 695 (1985).
4. A. Cezairliyan and J.L. McClure, A Microsecond-Resolution Transient Technique for Measuring Heat of Fusion of Metals: Niobium, International Journal of Thermophysics, in press.